Reducing the Size of the Borehole X-ray Fluorescence Spectrometer (XRFS) Probe: Preliminary Design Study

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Executive Summary

This report describes the recent work to design a borehole X-ray fluorescence spectroscopy (XRFS) instrument for the NASA Mars Subsurface Access Program. The instrument is to be used for elemental analysis of regolith strata in a pre-drilled borehole. The drills being developed now create a 30-mm diameter borehole; drilling a hole this size may require excessive power, so reducing the diameter of the borehole and probe would be a great advantage. This project began with a broad inquiry into the critical technologies for the components of the XRFS probe. The second part included a review and summary of the important considerations for the design of an XRFS borehole probe. Several preliminary designs were developed as the third component of the study. Finally, an evaluation of alternate sources for the primary X-rays was included. This evaluation covered the miniature X-ray tubes used in an existing probe and options for the proposed probe. We also surveyed the radioisotope sources used for previous space X-ray instruments, including a new type of X-ray source that has just become available commercially; a pyroelectric generator shows great promise.

The instrument being constructed under this NASA contract is based on a terrestrial XRFS sensor that was designed to detect metals in soils for *in-situ* analysis of heavy metal contaminants in the environment. The probe housing is 1.75 inches (44 mm) in diameter by 20 inches (500 mm) long. A sealed X-ray tube is used as the excitation source to provide adequate incident radiation to allow a spectrum to be taken in 100 sec with minimum detection limits of about 100 ppm for most heavy metals.

X-ray fluorescence operates by detecting characteristic X-rays emitted by the atoms in a sample. Both the minimum detection limits (MDL) and the ability to quantify elements are mostly dependent on the number of X-rays collected. The counts are limited by the data collection time, the intensity of the incident beam, and the geometry. For a 10-m depth and a vertical resolution of 1 cm, 1000 spectra must be collected for each hole investigated. The 100-sec acquisition time of the SCAPS terrestrial instrument would imply a total measurement time of about 28 hr per hole. Lower detection limits or more precise quantitative results may be achieved with longer acquisition times. A less intense X-ray source would also increase this total time. The source and detector must be protected from accumulating dust by some kind of window. The peaks from some elements can overlap, raising detection limits, making the determination of net intensities for the elements difficult, and (in extreme cases) interfering with identification of the elements. The X-rays from the excitation source may also have characteristic energies that can overlap with some elements.

All of the X-ray sources and detectors being considered are capable of operating at Mars ambient temperatures. The greatest challenge of the Mars ambient comes with the use of high voltage to generate incident X-rays to excite the sample for analysis. The breakdown resistance in Martian atmosphere is less than 1000 volts per centimeter. Consequently, some form of high voltage potting is necessary if an X-ray tube is used. X-ray tubes have been in service since early in the previous century and have undergone much development and optimization. Pyroelectric X-ray sources are relatively recently developed sources that show great promise. Radioisotope sources have the highest reliability and ruggedness of any X-ray source.

The first performance consideration is the capability of detecting most or all of the elements in the periodic table. The ability to accurately quantify a particular element is mainly limited by the measured precision of its X-ray emissions, which depends on both the number of X-rays collected and the background. The main performance metric is the MDL. Given the limitations on detectors and geometry, the performance depends almost exclusively on the strength of the primary X-ray source.

Over the last 10 years, several miniature X-ray tubes have appeared on the market. Given the size constraints imposed by the detector, the precise choice of X-ray tube is not a major factor in the size of the probe. The location of the high voltage power supply does have a considerable influence on the design.

Radioisotope sources have been the X-ray and particle sources of choice for all actively-excited spectrometers used to date in space applications. The fact that they require no external power and never fail are compelling reasons for their choice. However, the intensity of this type of source is weak and its use may imply unacceptably long data collection times. Cost is not a major factor in the choice of excitation source.

The smallest commercial off-the-shelf package for an energy-dispersive detector is 18 mm diameter by 40 mm long. A detector of this general type is used on the SCAPS sensor and has been included on every Mars rover to date. It is the obvious choice for the new borehole instrument. The size of this component is the main limitation to reducing the size of the probe.

Three design alternatives are described here. The first is the original design in the proposal for this contract with a diameter of 30 mm. The second design considers the new, miniature X-ray tubes that have come on the market. The diameter could not be reduced in this design because of the fixed size of the detector. The third design changes the arrangement of the detector to further reduce the diameter. The likely performance cost of this design will be a factor of two increase in minimum detection limits for sodium, with decreasing effects for the elements across that row in the periodic table

(through chlorine). There would be little or no monetary cost for this change and little risk.

There are no fundamental limitations to achieving a 10-mm diameter, but the detector module would require complete re-designed. The cost of the required development effort is estimated to be about $1M. Because there is no market pressure to further reduce the size of detectors, the cost would have to be borne by this project. There is, other than cost, relatively little risk for this effort because it is mainly re-arranging and re-packaging of the same components.

Introduction

This report describes the preliminary design effort for a borehole X-ray fluorescence spectroscopy (XRFS) instrument for the NASA Mars Subsurface Access Program. The original proposed design was 30 mm in diameter and was based on an existing terrestrial probe for analyzing heavy metal contaminants in soils. The new probe is intended to operate in conjunction with planetary drilling systems being developed for future missions in the next decade. Because the borehole will be pre-drilled, the new probe can be smaller than the existing probe and analyze a wider range of elements.

The drills being developed may require excessive power consumption to drill a 30-mm diameter borehole, so a smaller diameter for the new XRFS probe would be a great advantage. This study was added to the project to investigate whether the new probe could be made smaller and to determine development costs for a smaller probe. A multi-pronged approach was taken to obtain as much information as possible for future plans and decisions. The design of the prototype XRFS probe being built under this project will not necessarily be changed by this study, but it is being undertaken early in the project so that as much information as possible can be included.

The study began with a broad inquiry into the critical technologies for the components of the XRFS probe. The purpose was to find all appropriate components that were available off-the-shelf, to insure that no possible choices were overlooked, and to determine if any new developments were likely in the near future. Personal contacts of the APL-UW Principal Investigator (PI) and the NASA Technical Representative (TR) were employed to search for new vendors to manufacture these critical components and to discuss future developments with both familiar and new vendors. To broaden the investigation as much as possible, a formal search was made for potential vendors. A letter requesting information was sent to a large number of possible vendors.

The vendor search revealed no new developments or vendors of which the PI and the NASA TR were not aware. The investigation through personal contacts revealed two new companies that have begun to offer miniature X-ray sources recently. The personal relationships and reputations of the PI and the NASA TR were essential in obtaining from vendors the technical details necessary for evaluation of the components and development of the preliminary designs included herein. Many of these vendors were willing to discuss their products in phone conversations but were reluctant to reveal technical details in writing. This was especially true for possible future developments.

The second part of the study was a review and summary of the important considerations for the design of an XRFS borehole probe. Based on the experiences of

the PI and NASA TR in developing the existing terrestrial XRFS probe (by the PI) and from the extremely successful XRFS instrument deployed on the Viking Lander (by the NASA TR), this design review comprised a major part of this study. The general requirements for XRFS instrument performance as well as requirements specific to borehole deployment and to the Mars environment are included.

Several designs were developed as the third component of the study. These designs are only preliminary, showing the arrangement of the major components and how they would be assembled in the probe. The detail included is sufficient to evaluate whether the components would fit together and what the diameter of the final probe would be (within about 10%). Solid models of the designs were made to facilitate visualization, insure there were no mechanical conflicts, and determine geometric parameters to evaluate the performance of the various designs.

Finally, an evaluation of alternate sources for the primary X-rays was made. This evaluation covered the miniature X-ray tubes used in the existing probe and proposed for the new probe. It also included the radioisotope sources used for previous space X-ray instruments. A new type of X-ray source that has just become available commercially, the pyroelectric X-ray generator, was included in this part of the study because of its great promise.

(Existing) Terrestrial Probe

The instrument being constructed under this NASA contract is based on a terrestrial XRFS sensor that was designed to detect metals in soils at levels from about 100 ppm. The purpose of the sensor was *in-situ* analysis of heavy metal contaminants in the environment. A detailed description and results of bench and underground tests have been published[1]. Only a brief description will be given here. The sensor was built as part of the Department of Defense Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) program. A suite of sensors was developed that could be deployed via a direct-push penetrometer system. The sensor was named the SCAPS XRF Metals Sensor and will be referred to as the SCAPS sensor herein.

The SCAPS sensor was developed jointly by the U.S. Naval Research Laboratory and the U.S. Army Engineer Waterways Experiment Station. The effort was funded by the Strategic Environmental Research and Development Program (SERDP), the Environmental Security Technology Certification Program (ESTCP), and the U.S. Army Environmental Center.

The sensor consists of an above-ground electronics package located in the SCAPS truck, the below-ground probe, and an umbilical cable (Figure 1). The design considerations for this probe have been published[2]. The probe housing is 1.75 inches (44 mm) in diameter by 20 inches (500 mm) long. Mounted on a rail inside the probe are the X-ray source, detector, and the detector preamplifier. This rail also holds an X-ray collimator to direct the excitation radiation onto the soil and limit the fluorescence X-rays entering the X-ray detector. The radiation enters and returns through a robust X-ray transparent window in the probe housing. A sealed X-ray tube is used as the excitation source to provide adequate incident radiation to allow a spectrum to be taken in 100 sec.

The detector is a commercial unit packaged in a small case consisting of a silicon PIN diode with self-contained cooling, connected in close proximity to a low-noise preamplifier. The down-hole preamp provides sufficient signal to be driven through the umbilical cable and reduces noise pickup from the X-ray tube power leads. At the time no preamplifiers small enough to fit within the sensor housing were available and the unit was reassembled to fit. Off-the-shelf detector electronics provide the necessary power

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[1] W.T. Elam, J.W. Adams, K.R. Hudson, B. McDonald, and J.V. Gilfrich, Subsurface measurement of soil heavy metal concentrations with the SCAPS X-ray Fluorescence (XRF) Metals Sensor, *Field Analytical Chemistry and Technology* **2**, 97–102 (1998).

[2] W.T. Elam and J.V. Gilfrich, Design of an X-ray fluorescence sensor for the cone penetrometer, *Advances in X-ray Analysis* **38**, 699–704 (1995). Also W.T. Elam, R.R. Whitlock, and J.V. Gilfrich, Use of X-ray fluorescence for in-situ detection of metals, *Proc. SPIE* **2367**, 59–69 (1995).

supplies to operate the down-hole detector components and include a signal-shaping amplifier. An exhaustive review of commercially available X-ray power supplies at the time the sensor was designed revealed that none could function with the long umbilical cable. Consequently a custom supply that provides 30kV as well as filament current to the tube from a single electronics package was designed and fabricated at the Naval Research Laboratory (NRL). This power supply is also connected to a safety interlock system as required by government safety standards for terrestrial operation.

The X-ray window is boron carbide. Its diamond-like crystal structure provides strength and hardness. Boron and carbon are low atomic number elements that have low X-ray attenuation coefficients in the relevant energy range, allowing high X-ray transmission to excite the elements in the soil and return transmission of the fluoresced X-rays. The window is 0.5 inch (12.7 mm) in diameter and located 6.8 inches (172 mm) from the down-hole tip of the sensor.

The umbilical cable conducts the high voltage and filament power required by the X-ray tube, the electronics and cooling power for the detector and preamp, and the signal pulses from the detector. It is fully shielded both for noise immunity and high voltage safety. The 42-m umbilical cable used currently is theoretically expandable to 300 m.

TR 0503

Interlock

Umbilical

"fail-safe"
X-ray On
light

Detector
electronics

HV
power supply
(key switch)

X-ray Tube

Detector

Detector
Preamp

Window

Collimator
Assembly

MCA
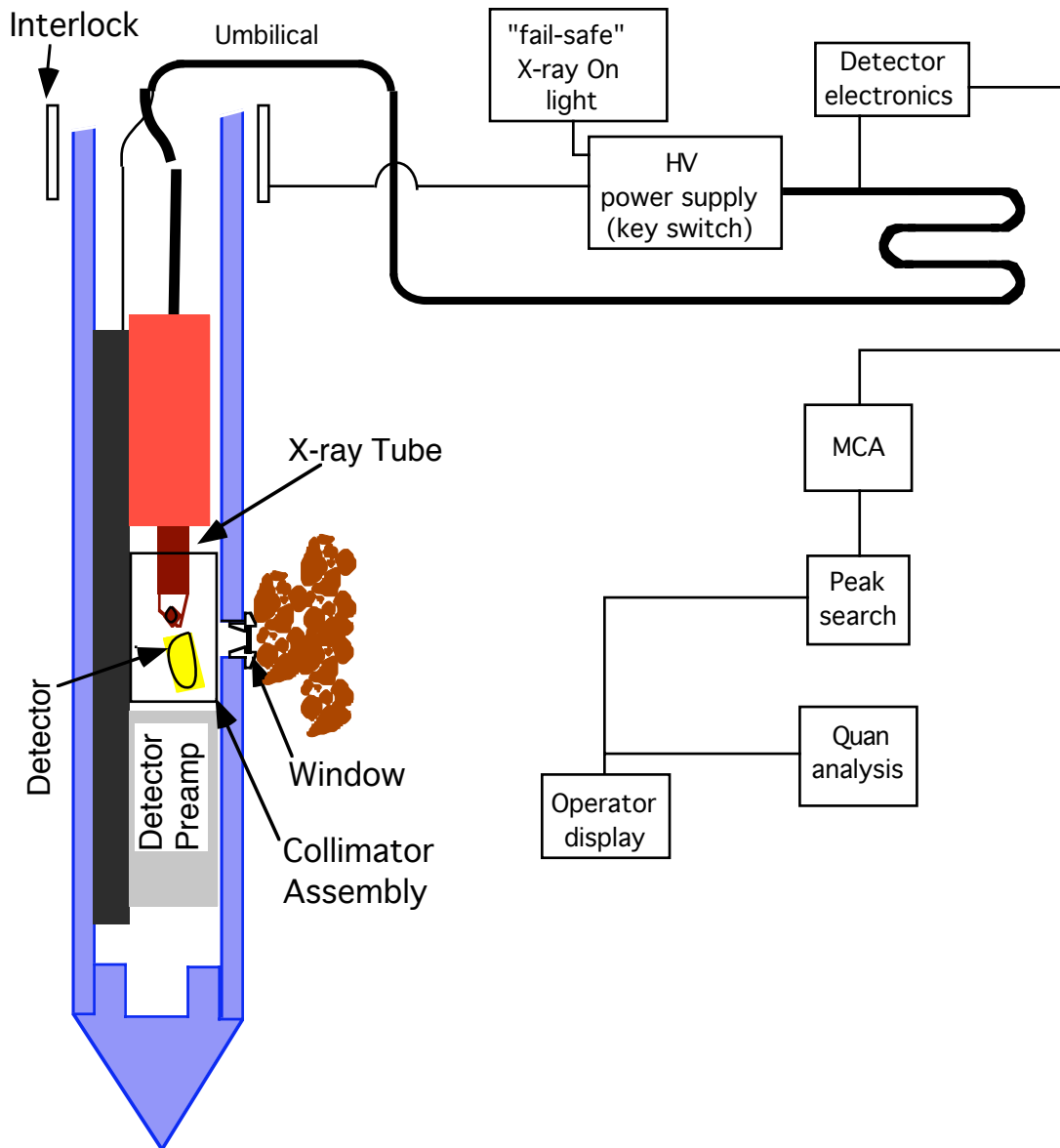
Peak
search

Quan
analysis

Operator
display

Figure 1. Schematic and engineering diagram of the SCAPS XRF Metals Sensor

Design Considerations

X-ray Fluorescence

General description

X-ray fluorescence operates by detecting characteristic X-rays emitted by the atoms in a sample (see, for example, Van Greiken and Markowicz[3] and Tertian and Claisse[4]). An X-ray source bombards a sample with incident X-rays, where the atoms are excited. The excited atoms then emit fluorescence X-rays that are detected as a function of the energy of the X-rays. The incident X-ray can have any energy that is greater than the binding energy of the core electron of the analyte atoms. The fluorescence X-rays are produced with a constant and well-known energy (different for each type of atom). The excitation of core states (unlike the valence states used in atomic and optical spectroscopy) implies that the fluorescence X-rays have relatively high energies and are independent of the chemical state of the atoms. The relatively high energy of the X-rays makes them penetrate any type of matter for distances of several microns to several millimeters, regardless of optical transparency. Any type of atom with sufficiently energetic core levels can be detected in any matrix. Generally, elements with atomic number 10 (sodium) or greater can be detected under ambient conditions.

Borehole-specific requirements

To understand the specific requirements for a borehole instrument, a scenario for its operation is helpful. In the simplest case the probe will be lowered into a borehole after the drill string is removed to take measurements during descent. Measurements will be made at discrete depths, preferably close enough together to identify separate strata and obtain elemental information about each of them. The probe may be inserted after the bore is complete, or it may be sent down at periodic intervals during drilling, such as when a core is removed. For a 10-m depth and a vertical resolution of 1 cm, 1000 spectra must be collected for each hole investigated. The 100-sec acquisition time of the SCAPS terrestrial instrument would imply a total measurement time of about 28 hr per hole. The choice to achieve lower detection limits or more precise quantitative results, or the forced choice of using a less intense X-ray source, would all increase total measurement time. Beyond a certain point a practical limit is reached.

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[3] R.E. Van Grieken and A.A. Markowicz, *Handook of X-ray Spectometry, 2nd Ed.* Marcel Dekker, New York (2002).
[4] R. Tertian and F. Claisse, *Principles of Quantitative X-ray Fluorescence Analysis*, Heyden, London (1982).

The walls of any borehole are not smooth and have variations on the order 10% of the diameter. The window of the borehole probe thus cannot be held in contact with the regolith material being measured and cannot even be held a reliable distance away within a few millimeters. The data acquisition and analysis methods must be able to account for this variation and still yield acceptable quantitative results. This can be accomplished by making the geometry as insensitive to distance variations as possible and by using scattered radiation as a calibration input to compensate for distance changes. Given the low attenuation of the Mars atmosphere, the variations in distance will mainly cause changes in the overall signal strength, which does not affect relative intensity for the different elements.

Spectrum

Line overlap and resolution

The most important considerations for elemental identification and quantification are the ability to determine the energy and intensity of the emitted X-rays. The spectrum from a typical instrument has several peaks for each element. The width of the peaks in energy-dispersive spectroscopy is dominated by the detector resolution. (Wavelength-dispersive spectroscopy has much better energy resolution but its bulk, complexity, and reduced sensitivity make it inappropriate for this application at its current stage of development.) The peaks from some elements can overlap depending on the detector resolution. It is important to consider that these overlaps, which can raise detection limits, can make the determination of net intensities for the elements difficult, and (in extreme cases) can interfere with identification of the elements. Energy-dispersive detectors are available in several types, but only the semiconductor detectors have sufficient resolution to allow reliable separation of the majority of elemental peaks. Typical resolution for this type of detector is 150 eV when cooled to about –100ºC. Such cooling originally required liquid nitrogen but can now be achieved by Peltier (electrical) cooling. Several detectors based on PIN diodes and silicon drift diodes are available in sealed packages with internal cooling.

The X-rays from the source may also have characteristic energies that can overlap with some elements. These overlaps must also be considered in the choice of source.

Flux and collimation

Because individual X-rays are counted and analyzed by the detector, the principal noise in an XRF spectrum is the variation in counts from the Poisson emission statistics of X-rays. This means that both the MDL and the ability to quantify the amount of each element are mostly dependent on the number of X-rays collected in the spectrum. This implies that the counts in the spectrum should be as large as possible. The counts are

limited by the time available to accumulate the spectrum, the intensity of the incident beam exciting the fluorescence, and the solid angles of incident and detected beams.

Both the incident beam and the X-rays reaching the detector must be collimated for two reasons. First, no fluorescence from regions other than the sample should enter the detector, as this would cause detection of elements not present in the sample as well as cause extraneous scatter. Second, the resolution of semiconductor detectors is best in a region near the center of the active detection volume and degrades for X-rays that impinge near the edges of the active volume. Proper collimation can improve resolution significantly.

The counting and analyzing of individual X-rays in the detector has a secondary effect. The detector and electronics require a certain amount of time to process each X-ray. During this processing time, the detector is either blind to additional X-rays or confuses the two X-rays as one, resulting in a count at the sum of the energies. Modern digital pulse-processing electronics has algorithms to reduce these effects, but they are still the main limitation to the count rate. The count rate in the detector thus has severe limits and can affect the energy resolution well below the limit. At extremely high count rates the detector saturates and the signal actually decreases.

As long as the source is sufficiently powerful, the spectrometer can be operated near the upper limit of the detector count rate, with collimation optimized for spectral purity. The count rate will depend, of course, on the amount of fluorescence emitted by the sample, so an adjustable source is desirable for optimum performance. However, for weaker sources some compromises must be made and the time to acquire a spectrum may have to be extended.

Attenuation and scatter

Optical elements in the beam between the source and the sample, and between the sample and the detector, will both attenuate the beam and scatter the X-rays. If scattered X-rays enter the detector, they produce additional background. (The main background is from incident X-rays scattered by the sample.) The attenuation decreases the signal, and is particularly important for the lighter elements (like sodium) that emit low energy characteristic X-rays. The source and detector both have windows (almost always beryllium because of its low attenuation). These windows are the limiting factor for detection of the lighter elements. A typical thickness for the detector window is 13 microns (0.0005 inch), which produces an attenuation of about 50% at the sodium emission line (Na K-L2,3 or Kα at 1041 eV). The geometry and collimation are typically arranged so that the scatter from the source and detector windows does not enter the detector. The detector window is necessary to provide reduced pressure to avoid heat transfer and allow the detector to be cooled as described above. The source is sealed to

provide the high vacuum necessary for X-ray tube operation, the correct gas pressure for pyroelectric generation, or to prevent escape of the radioactive material for a radioisotope source.

Physical protection

The source and detector must be protected from dust accumulation by a window, so some attenuation and scatter are unavoidable. This window should be as thin as possible but still sufficiently rugged to survive the treatment the instrument will receive. Window design is one of the most critical compromises in the instrument design because it is directly in the beam and in front of the sample. It is also the main protection for the instrument. Its failure will often render the instrument inoperable, as with the terrestrial probe operating below the water table. One advantage of the Mars ambient is its dryness, so incursion of water is not a potential problem. Furthermore, the low pressure of the atmosphere offers relatively little attenuation to X-rays, making the geometry less restrictive. The main consideration for a Martian borehole probe is protection from Martian dust, both from drifting dust and that produced by the drilling operation. It must be rugged enough to survive descent into the borehole and not be punctured by irregularities in the walls or by debris. The window can be recessed, which allows it to be more fragile and thus thinner (which provides less attenuation and scatter). The recess must be carefully designed not to collect dust, which would produce an interfering signal.

Mars Ambient

Atmosphere

The Martian atmosphere has an ambient pressure of about 8 millibars (0.8 kPa) with variations of only about +/– 15%. It consists of mostly carbon dioxide with a few percent of nitrogen and argon. Other components are less than 0.5%. X-ray attenuation in pure carbon dioxide at this pressure is only about 2% per cm at 1 keV, so atmospheric attenuation of the relevant X-rays is negligible.

The greatest challenge of the Mars atmosphere comes with the use of high voltage to generate incident X-rays to excite the sample for analysis. In order to excite a wide range of elements in the periodic table, the K lines of Na (1041 eV) through Zr (15700 eV) and the L lines of Cd (3130 eV) through Pb (10500 eV) must be excited. This requires incident X-rays with more than 18 keV. To efficiently excite X-rays of this energy, an over-voltage of about 2.5 times is optimal. Voltages of 30 to 40 kV are typically used.

The breakdown voltage in gasses is described by Paschen's law to within the accuracy needed here[5]. This law predicts that the minimum breakdown resistance is reached at or near Martian ambient pressures in carbon dioxide. The breakdown resistance at this minimum is less than 1000 volts per centimeter. For 30 to 40 kV, the distances required to reliably avoid breakdown are prohibitive in a borehole instrument. Some variation from this minimum is seen in measurements on a gas mixture that more closely approximates the Mars atmospheric composition[6]. However, these minor deviations are not sufficient to allow open, high voltage connections in an instrument whose size is under the constraints in this study. Consequently, some form of high voltage potting is necessary if an X-ray tube is used.

Temperature range

The borehole instrument must be able to operate over the temperature range on Mars (zero to –100ºC). Methods for constructing electronics to operate in this range are well established, but the borehole instrument poses a few specific challenges. Potentially the most difficult is the high voltage insulation. Potting compounds become stiff and often fracture at very low temperatures, leading to failure. Dow Corning® 3-6121 Encapsulating Elastomer is rated for temperatures below –65°C, so it is a good candidate. It has higher viscosity than the Sylgard® 184 Silicone Elastomer used for the SCAPS sensor, so more sophisticated fabrication techniques like vacuum injection will have to be used. All of the X-ray sources being considered are capable of operating at Mars ambient temperatures provided they are mounted such that thermal expansion does not distort or damage them. All except the radioisotope sources generate some heat (typically a watt or so), so they may be warmer than ambient while operating.

Reliability

Any instrument for extra-terrestrial operation must be able to operate for an extended lifetime with no human intervention. This places strict reliability requirements on all systems and components. X-ray detector systems similar to the ones that will be used in the borehole instrument have been used on prior Mars rovers[7]. The use of high-reliability electronic components will bring the majority of the probe systems up to the standard for non-safety-critical flight components. However, two of the X-ray sources being considered have not been used in space to date: X-ray tubes and pyroelectric sources.

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[5] E. Nasser, *Fundamentals of Gaseous Ionization and Plasma Electronics,* Wiley-Interscience, New York (1971).
[6] http://empl.ksc.nasa.gov/CurrentResearch/Breakdown/Breakdown.htm, verified Dec. 13, 2005.
[7] http://nssdc.gsfc.nasa.gov/database/MasterCatalog?sc=MESURPR&ex=1, verified Dec. 19, 2005.

X-ray tubes have been in service since early in the previous century and have undergone much development and optimization. They are routinely used in medical equipment and have proven robust and reliable. Moreover, they are similar in construction to microwave vacuum tubes (such as traveling wave tubes) that are routinely used in communications satellites. The techniques for potting and glass envelope fabrication to make rugged X-ray tubes are well developed. Many of the new, miniature X-ray tubes are of ceramic construction, making them even more rugged.

Pyroelectric X-ray sources are relatively recently developed sources that show great promise. They have the advantages of very low power and light weight. Their reliability and ruggedness are as yet unknown. The techniques necessary to improve their reliability and ruggedness are just being explored, and whether they will be sufficient for flight use remains to be seen. If so, they will be a valuable addition to the instrument choices.

Radioisotope sources have the highest reliability and most extreme ruggedness of any X-ray source. This accounts for their use in all extra-terrestrial X-ray spectrometers to date and makes a powerful argument for their use. However, they may not be adequate for a borehole instrument that must take spectra in a limited time during descent through the borehole and during drilling operations. In addition, the need to take spectra with depth resolution sufficient to identify strata may rule out radioisotope sources.

Instrument Performance

The purpose of this instrument is elemental analysis of regolith strata in a pre-drilled borehole to investigate the subsurface of Mars and possibly other bodies within the solar system. As such the primary performance criterion is the ability to quantify the elements present in a particular stratum in an acceptable time and with sufficient accuracy to obtain useful scientific information. For the purposes of this study, only relative performance will be considered; the detailed performance of the sensor will be evaluated by measurements on the actual prototype and covered in a later report.

The first consideration is the range of elements that the sensor can detect and quantify. Here only X-ray sources and detectors that are capable (in principle) of detecting most or all of the elements in the periodic table are considered.

The ability to accurately quantify a particular element is mainly limited by the precision with which its X-ray emissions can be measured. This is determined by the statistical variations in X-ray intensity due to the Poisson nature of their arrival times. In a given time interval the number of X-rays that are detected has an intrinsic variance (the

square of the standard deviation) equal to the number of X-rays. This means that the relative standard deviation is one over the square root of the number. For a given geometry and sample composition, the number of X-rays detected from a particular element is proportional to the source strength and the measurement time.

Detecting an element depends on both the number of X-rays collected from that element and the background present even in the absence of that element. Because the background is also subject to the same variations, the MDL is usually taken as three times the standard deviation of the background (converted to elemental concentration by an appropriate calibration coefficient). This is equal to three times the square root of the background counts in the spectrum. Both the desired signal and the background are proportional to the source strength. The background arises from scatter from the continuum of an X-ray tube and the detector peak-to-background ratio. These contributions are of comparable size for the X-ray tubes and detectors considered in this study. For a radioisotope source, there is no continuum, but the detector background from scatter of the characteristic X-rays is comparable to the continuum scatter from an X-ray tube. Consequently, the MDL depends almost exclusively on the strength of the primary X-ray source and its energy (which determines the efficiency with which a given element is excited).

The main performance metric is the MDL. Improvements in the ability to detect an element automatically imply improvements in the ability to quantify the amount present. Though there are some subtleties in this that will be considered in the final instrument design, the performance is dominated by the number of X-rays present in the spectrum, which is dominated by the source strength given the constraints on geometry and the available detectors for this instrument.

Sources

X-ray Tubes

X-ray tubes operate in much the same way as the original hot cathode tubes invented by Coolidge in 1913 (Figure 2). Electrons from a filament are accelerated by a high voltage and strike an anode. The energetic electrons excite atoms of the anode, which then emit their characteristic X-rays. Because the emitted X-rays must have sufficient energy to excite the analyte elements in the sample, the anode is chosen to emit X-rays of about 20 keV. Typical anodes are silver, rhodium, and molybdenum. Other anodes are employed for specific applications. Almost all laboratory analytical instruments use a rhodium anode because it excites most of the elements in the periodic table reasonably well and is rare enough that it seldom causes any interference with desired analyte elements. Most hand-held instruments use a silver anode because of its lower cost and because it efficiently excites (and does not interfere with) platinum group metals (including rhodium). Hand-held units are not usually used for precious metal analysis in jewelry because they lack the necessary precision, but they are often required to identify the very lucrative platinum group metals in scrap metal recycling.
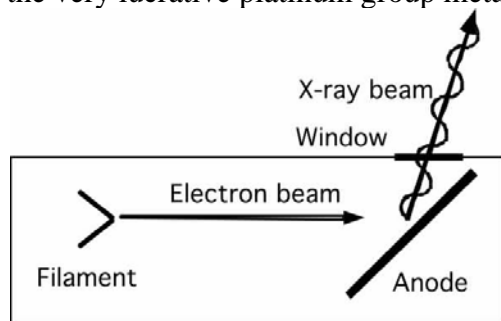
Figure 2. X-ray tube diagram

The electron source is almost always a heated filament made of a tungsten wire that gives off electrons by thermionic emission. The filament is resistively heated by passing a low-voltage current through the wire. The electron emission current is regulated by adjusting the filament heating power based on feedback from the output current of the high voltage power supply. The electron emission and acceleration must occur in a high vacuum, so the X-ray tube is constructed in a metal and insulator housing with a thin window through which the X-rays can escape. The window is usually beryllium for spectroscopic applications because of its ability to withstand atmospheric pressures at thicknesses that are transparent to the X-rays of interest. The vacuum also prevents breakdown inside the housing due to the high voltage. Electron energies of two to three times the characteristic X-ray energies of the anode are required to efficiently excite the anode. Accelerating voltages of 30 to 50 kV are typical in XRFS.

The spectrum of the X-ray tube is determined mainly by the choice of anode and by the accelerating voltage, and secondarily by the exit window material and thickness. In addition to the characteristic emission, the electrons excite a continuous spectrum called bremßtrahlung or "braking radiation." It is produced by deceleration of the electrons in the Coulomb field of the anode atoms. This radiation is the main source of the background in XRFS and its spectrum and intensity are functions of the anode atomic number and the accelerating voltage. The characteristic emissions can be reduced or eliminated by lowering the accelerating voltage, giving some control over the spectrum. If multiple series of emission lines are available from the anode, then they can be excited selectively by choosing the proper voltage. For example, a silver anode has K series emission lines at about 22 keV and L series emission lines at about 3 keV. The K series can be used to excite heavier elements and the L series to excite lighter elements. By using an accelerating voltage well above 3 kV and below 22 kV, the L series only is produced and the lighter elements are preferentially excited.

Figure 3. Miniature X-ray tube[8]

Over the last 10 years, several miniature X-ray tubes have appeared on the market. A typical example (Figure 3) has an outer diameter of 3/8 inch (9.5 mm) and is about 1.25 inches (32 mm) long. Similar tubes are available from several vendors, although some vendors do not sell the X-ray tube without their own housing, which is too large (1.3 inch or 33 mm) for the size constraints of the borehole. The tube shown in Figure 3 is a transmission-target design, which implies that the X-rays are emitted axially from the end of the tube. This geometry is not ideal for a borehole instrument. Other vendors offer side-window tubes like that used in the SCAPS sensor and are the likely choice for the new instrument. One vendor offers a line of X-ray tubes with a diameter of ¼ inch (6.4 mm). Given the size constraints imposed by the detector, the precise choice of X-ray tube is not a major factor in the size of the probe.

The component that does have a considerable influence on the size of the probe is the high voltage power supply for the X-ray tube. This component is also the main

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[8] Newton Scientific Inc., 255 Bent Street, Cambridge MA 02141. X-ray Module Specification NSI-2, Cygnet Miniature X-ray Source (2005). http://www.newtonscientificinc.com/, verified Dec. 23, 2005.

challenge for reliable operation under Martian ambient conditions. For the SCAPS sensor, this component was not included in the down-hole probe. This required having a 30-kV cable as part of the umbilical. High voltage power supplies that are small enough to fit within the 30-mm diameter are now available, but the choice of location for this component remains a significant challenge.

Pyroelectric generators

Pyroelectric X-ray generators, pioneered by Brownridge[9], are a relatively recent development in X-ray production. They have been commercialized and are available off-the-shelf for some applications. The main advantages of this type of source are its low power consumption and simplicity. Potential disadvantages are very low generated intensity and unknown reliability and ruggedness.

Pyroelectricity is a phenomenon whereby a transient voltage is generated in a crystal undergoing a temperature change. Under certain conditions electrons can be emitted by the crystal with several tens of keV of energy. If these electrons are directed at a suitable anode, characteristic X-ray emission will be generated in the anode similar to the X-ray generation in an X-ray tube. A very simple X-ray source can be constructed using a pyroelectric crystal, a resistance heater, an anode, and a suitable housing[10]. For optimal generation of electron beams, a low pressure gas is required, so an X-ray source is typically sealed into a small unit with a Be window.

An example commercial pyroelectric X-ray generator (Figure 4) has a housing diameter of about 13 mm, so the device is small enough to fit in a borehole instrument and may enable significant size and power reductions. If this type of device can be made sufficiently reliable and rugged to be useful in a flight instrument, it could be a very valuable component for X-ray spectrometers. One of these devices has been purchased by NASA as part of this project and is being evaluated.

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[9] J.D. Brownridge. Pyroelectric X-ray generator, *Nature* **358**, 287–288 (1992).

[10] J.D. Brownridge and S.M. Shafroth. X-ray fluoresced high-Z (up to Z=82) K x-rays produced by $LiNO_3$ and $LiTaO_3$ pyroelectric crystal electron accelerators, *Appl. Phys. Lett.* **85**, 1298–1300 (2004).

Figure 4. Pyroelectric X-ray generator[11]

Radioisotopes

Radioisotope sources have been the X-ray and particle sources of choice for all actively-excited spectrometers in space applications. The facts that they require no external power and never fail are compelling reasons for their choice. However, the intensity of this type of source is determined by the amount of source material and its specific activity, which is determined by its half-life and the purity of the emitting isotope. Isotopes with short half-lives have a high specific activity and make intense sources. However, the short half-life implies that the source will decay quickly. In practice, the time between preparation of the source prior to launch and its use (dominated by the time in transit) sets a limit to the minimum useful half-life. The competing reactions producing the isotope and their chemistry determine which isotopes can be isolated with sufficient purity to make practical sources. Furthermore, the X-rays emitted by the isotope must have an energy that is convenient for exciting the elements being analyzed. The source excitations should not place undue limits on the elements that can be detected by the instrument. When all of these limitations are considered, there are only a few isotopes that are useful for an instrument of this type.

Isotope	Half-life (years)	X-rays emitted	X-ray energy	Specific activity
Fe-55	2.7	Mn K	5.9 keV	2.2×10^3 Ci/gm
Cd-109	1.3	Ag K	22 keV	2.6×10^3 Ci/gm
Am-241	432.2	Np L	13.9 keV	3.2 Ci/gm

The X-ray energy of radioisotope Fe-55 is too low to excite much of the periodic table, so it is used only for limited purposes in XRFS instruments. Cd-109 is a frequent choice for hand-held units where the source can be replaced periodically. The only practical isotope for a borehole instrument with a long useful life on Mars is Am-241, which emits Neptunium L X-rays at 13.9 keV (42%) and a gamma ray at 59.5 keV (36%). The remainder of its disintegrations emits an alpha particle that can be used to

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[11] http://www.amptek.com/coolx.html, verified Dec. 19, 2005.

excite other X-rays in a secondary target. Both direct and secondary emissions were considered for this study.

Two vendors were contacted and asked to provide feedback about producing an Am-241 source suitable for this type of instrument, either from their available product line or as a custom design. A sketch of the desired source shows a direct source that uses the Np X-rays produced directly by Am-241 and an indirect source that uses the X-rays from foils excited by the alpha particles emitted by Am-241 (Figure 5). In this source a choice of several foils is included to provide three different excitation energies. This gives some flexibility in the excitation spectrum to make excitation of various elements more efficient and avoid interferences.
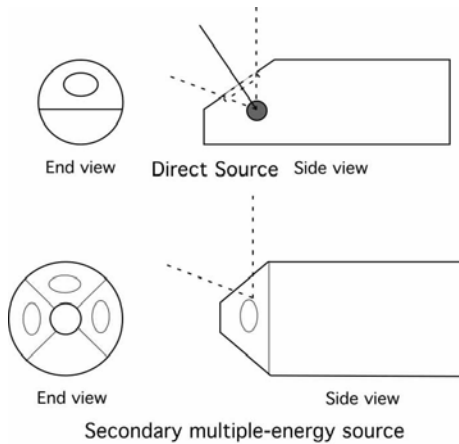

Figure 5. Diagram for source using Am-241

The X-ray tube in the SCAPS sensor has a measured emission of 4×10^8 photons per second per steradian in the characteristic line. This serves as a reliable order-of-magnitude estimate for the source strength required to achieve similar performance in a borehole instrument. Accounting for the full solid angle and the fraction of X-ray emissions per disintegration, a source of about 10^{10} disintegrations per second is required. This corresponds to 0.3 Curies (1×10^{10} Becquerel). This is a rather strong source, as the usual source strength for research purposes in measured in milliCuries (or MegaBecquerel). Industrial sources of this strength for both Am-241 and Cd-109 can be prepared and require less than one gram of material. The usefulness of an Am-241 source of this size will be limited by the penetration distance of 3.9 keV X-rays, which is a few micrometers, and by the penetration distance of the alpha particles, which is about the same due to their much higher energy. Source arrangements that spread the source volume over a disc or hemisphere will be necessary to achieve the desired output from this size of source. Lower activity sources can be used with consequent degradation in performance.

Detectors

Silicon-diode energy-dispersive X-ray detectors are the mainstay of portable XRFS instrumentation. They operate by producing a current pulse in the active region of the diode that is proportional to the energy of the absorbed X-ray. A reverse voltage bias on the diode both insures that there is no forward conduction in the diode and sweeps the generated charge cascade to the electrodes before recombination. Modern detectors, unlike their earlier counterparts, are made with intrinsic silicon of extremely high purity. They are stable over a wide temperature range and can be thermally cycled an indefinite number of times. They must be cooled to about –50°C for noise reduction, but this is accomplished by electrical Peltier coolers that are integral to the detector. A detector of this general type is used on the SCAPS sensor and has been included on every Mars rover to date. It is the obvious choice for the new borehole instrument.

Figure 6. Commercial energy-dispersive X-ray detector[12]

The smallest commercial off-the-shelf package for an energy-dispersive detector has dimensions of 18 mm diameter by 40 mm long (Figure 6). The X-ray entrance window is on the metal can at the left. This metal can contains the actual detector diode, the Peltier cooler, and the first stage of amplification. These dimensions are the current most stringent limitation to reducing the size of the borehole probe. There is one alternate vendor for this type of detector, but they were not willing to give any dimensions for a custom configuration without a specific set of requirements and a request for quotation. The dimensions are similar to the unit shown. Further reduction in the size of the detector package will only be achieved with a significant development effort, the cost of which would be borne by this project.

The performance of this type of X-ray detector has three components: resolution, efficiency, and count rate. The measurement of the pulse height has an uncertainty from electronic noise and an intrinsic component due to the partially-random nature of the

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[12] http://www.amptek.com/oem.html, verified Dec. 23, 2005.

charge cascade. This uncertainty causes a spread in pulse heights for a given energy of X-ray, giving a width to the peaks for each element in the spectrum. The efficiency with which the unit detects X-rays depends on the transmission through the window that seals the module, absorption in a dead layer at the surface of the diode, and the thickness of the active layer. The dead layer in modern detectors is small enough to have negligible effect. The efficiency at low X-ray energies is dominated by transmission through the window. A 0.5-mil (13 μm) Be window transmits 25% of the X-rays at 1 keV of energy (the sodium K lines). The efficiency at high X-ray energies is limited by X-rays passing through the active region without being absorbed. A typical active region is 0.5 mm thick and absorbs about half of the incident X-rays at 20 keV. Achieving adequate resolution requires some time (typically a few microseconds) for pulse processing to average out the electronic noise. The resolution degrades at high count rates due to overlap of X-ray arrivals or shorter pulse processing time in adaptive processors. Modern detectors have maximum count rates of several thousand counts per second.

One disadvantage of these smaller detectors is their peak-to-background ratio. Not all of the charge pulse produced by an absorbed X-ray is collected during the pulse processing time. The remaining charge shows up as a background throughout the spectrum. This background limits the ability to detect small amounts of an element, and while it can be reduced by appropriate detector construction techniques, it is close to the limits that can be achieved within the constraints of the construction materials and size of the units. Si-PIN diodes typically have better peak-to-background ratios than silicon drift diodes.

Design Alternatives

Several alternative designs were considered and some solid models were developed to insure that the components would fit together and to better determine the minimum diameter possible. Unfortunately, no new components or developments were discovered that could make the sensor dramatically smaller in the near future.

The first is the original design in the proposal for this contract. The second design considers the new, miniature X-ray tubes that have become available. The diameter could not be reduced in this design because of the fixed size of the detector. New or different detectors were not considered because none are available that meet the requirements of this instrument. Because the detector was the limiting component in the second design, the third design changes the arrangement of the detector to further reduce the diameter.

Design I – based on the SCAPS XRF Metals Sensor (Dia. 30 mm)

The original design concept on which the proposal to build this instrument was based is copied from the proven design of the SCAPS sensor. A new solid model of the existing device was constructed from the actual unit at APL-UW (Figure 7).
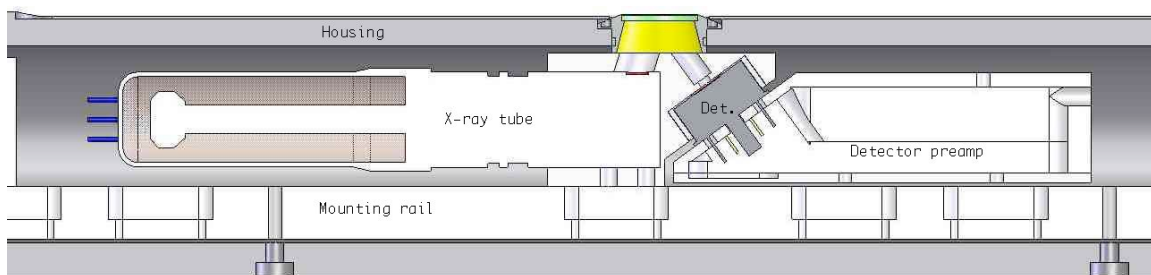

Figure 7. Design I based on the SCAPS XRF Metals Sensor (Dia. 30 mm)

The inside diameter of the housing is 1.375 inch (34.9 mm) and the mounting rail is 0.373 inch (9.5 mm) thick. Thus the major components fit within a diameter of 1 inch (25 mm). Because the new instrument will be deployed in a pre-drilled hole, the housing can be much thinner. Also, the very rugged window (in green at the top center of Figure 7) will not be needed for a pre-drilled hole. Some kind of housing is required and a window to protect the components and the analytical volume from dust will be necessary. The additional room for the housing, the window, and the beam path set the diameter of this design at 30 mm.

The major advantages of this design are its proven performance, with several years of use (and abuse) during field work at closed military bases[13]. MDL are near 100 ppm for most heavy metals in 100 sec of measurement time. This has been verified by repeated calibrations and holds for two different prototypes. The sensor has been licensed and is in commercial production[14]. Another advantage is that the beam path geometry is the least sensitive of the designs to distance between the sample and the window. The beams are mostly perpendicular to the housing axis, causing smaller changes in the excitation volume with distance.

This design does have significant disadvantages. The X-ray tube is 0.75 inch (19 mm) in diameter and the necessary room for high voltage insulation results in a diameter that cannot be reduced much below 30 mm. This tube also requires a fairly large filament power of about 2 watts. This significantly raises the minimum power consumption. Finally, this is a side-window X-ray tube. This, combined with the large diameter, makes the beam path angles difficult to fit within a diameter any smaller than 30 mm.

Design II – using new off-the-shelf components (Dia. 30 mm)

Several miniature X-ray tubes are now available commercially that are significantly smaller than the one used in the SCPS XRF Metals Sensor. The advantages of a design with a miniature X-ray tube (Figure 8) are the lower power consumption for the filament and the possibility of including the high voltage power supply in the down-hole housing. The filament power for these new miniature X-ray tubes is about 0.5 watt, a factor of four improvement over the tube used in the SCAPS sensor and a savings of 3.5 watts. High voltage power supplies with a diameter of less than 30 mm are available from at least two vendors. Having this supply down-hole reduces the complexity, size, and environmental constraints on the umbilical cable. The lack of a high voltage cable in the umbilical makes it much easier to fabricate to withstand the Mars ambient temperature and remain flexible. The smaller size of the umbilical is also an advantage in automated handling during probe deployment down the borehole.

[13] S.H. Lieberman, P.A. Boss, J. Cortes, and W.T. Elam, "Site Characterization and Analysis Penetrometer System (SCAPS) Heavy Metal Sensors Demonstration/Validation", SPAWAR Systems Center San Diego, Technical Report 1868, Dec. 2001.
[14] http://www.austinai.com/conepenetrometer.html, verified Dec. 23, 2005.
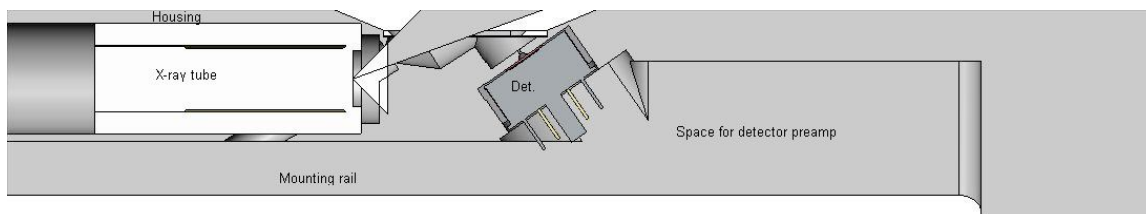
Figure 8. Design II with end-window X-ray tube (Dia. 30 mm)

The disadvantages of Design II are not a particular problem at the prototype stage. The new X-ray tubes do not have a long performance record, so their reliability and ruggedness are not as well proven. They were developed and are routinely used, however, for handheld XRF instruments, so neither should be a problem. The performance of this design is also unproven but is expected to be as good as Design I because the output of these X-ray tubes is comparable. Two of these tubes from different vendors are being obtained for evaluation of output and ruggedness.

The design (Figure 8) has an end-window X-ray tube, where the X-rays exit along the tube axis within a cone of about 45 degrees half-width. This arrangement was used to show that this type of tube could be used. Some tubes are available only in end-window configuration, while others are available with end or side windows. A side-window tube could easily be incorporated into Design II.

One consideration for this design is that the beam angles are steeper with respect to the housing axis. This means that variations in the distance from the housing to the sample (the borehole wall) will change the overlap between the excitation beam from the X-ray tube and the volume viewed by the detector. This can be compensated somewhat by using a larger excitation beam and making the analysis volume limited by the detector collimation. There is little or no cost for this since the X-rays from the tube are emitted into a large cone. It may have minor implications for the size of the protection window in the housing.

Design III – using a perpendicular detector arrangement (Dia. 22 mm)

To realize a substantial reduction in probe diameter, another design was developed with the detector aligned along the housing axis (inspired by the arrangement of the end-window X-ray tube in Design II). This allows the detector to be positioned so that only its minimum diameter impacts the probe dimensions. The fluorescence X-rays from the sample impinge on the detector at an angle, which will hurt the efficiency at low X-ray energies because of the longer path through the Be window and dead layer. This effect will reduce the transmitted X-rays by almost a factor of 2 at 1 keV. This will raise the detection limit for sodium by about 25% and have a smaller effect on heavier elements. The effect will be minimal above about 1.5 keV (the K lines of aluminum).

However, the longer path through the active region of the detector will improve the efficiency at high energies by a similar amount.

The big advantage of this design (Figure 9) is the smaller diameter of about 22 mm. The filament power is again about 0.5 watt, retaining this power savings.
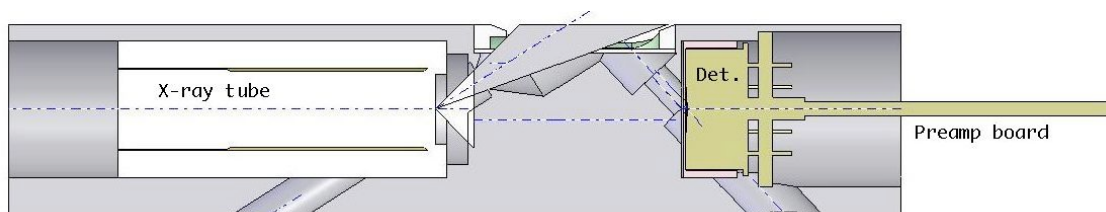

Figure 9. Design III with a perpendicular detector arrangement. (Dia. 22 mm)

This design is unproven and the presently available high voltage power supplies cannot be included in this diameter. However, a custom-fabricated supply might be possible and at least two vendors are working on smaller high voltage supplies, so it may be possible in the near future. The design is expected to provide comparable performance to designs I and II because it also uses an X-ray tube and the compromises necessary to mount the detector along the axis are not severe. A side-window X-ray tube could be used in this design with little modification provided that it was of the new miniature type. The use of the larger, well-proven X-ray tube from the SCAPS sensor is not possible in this reduced diameter.

This new design could be employed for the new sensor being built under this contract, or for a next-generation instrument. Although unproven, it is certainly appropriate for the prototype stage. Its reliability and ruggedness are expected to be about the same as any design using a new X-ray tube, because these properties are mainly due to the tube and not to the geometry. Within the class of new, miniature X-ray tubes this design can be adapted to different tubes by modifying the housing.

This design also is more sensitive to variations in the distance from the housing to the sample because the detector viewing angle is fairly steep. Again, using a larger excitation beam so that the analysis volume is limited by the detector collimation compensates for the steep angle. It is more difficult in this geometry and will consequently be less successful, but it is not expected to have a major impact on quantitative performance.

Unfortunately, feedback from the detector manufacturer indicated that this design was not useable. The entrance angle for the detector was not compatible with the internal arrangement of the detector. It has a collimator to prevent X-rays from striking the edge of the active element. The performance at the steep entrance angle would be unacceptably degraded. This design, however, had significant influence on the final design.

Choice of excitation source

Because the detector is the size-limiting component at present, the choice of excitation source can be made on the basis of performance, complexity, reliability, and power consumption.

The clear winner for simplicity, reliability, and power consumption is the radioisotope source. Construction of a radioisotope source with performance comparable to an X-ray tube is possible but challenging. The safety and regulatory requirements impose further challenges with this type of source. Cost is not a major factor. Rough estimates from two vendors indicate that such a source will cost a few tens of thousands of dollars. If a source of the required strength cannot be obtained, then performance will be degraded. This will result in longer measurement times. A reduction of at least an order of magnitude and possibly two or more in source strength may be mandated. This will make the measurement times very long and will probably prohibit any stratigraphy in the borehole. On the plus side, a radioisotope source can be incorporated into the design at almost any stage because it requires no power or cabling and can be made very small.

The pyroelectric X-ray generators are intermediate between the radioisotopes and X-ray tubes in terms of simplicity and power consumption. They are a new and completely unproven source that have only had feasibility demonstrations done for a few applications[15]. In the example cited, data collection times of 1000 sec were used and elements at concentrations of several hundred ppm could not be detected. Nevertheless the units are commercially available, have withstood the rigors of the marketplace, and are starting to be used more widely. The intensity of the current products is rather low, and performance would be correspondingly poor. The precise details can be better evaluated once the spectrum of an evaluation unit has been measured. Future developments of this device should be watched carefully, as it will prove very valuable if it goes into routine usage and receives further development effort.

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[15] H. Ida and J. Kawai, Identification of glass and ceramics by X-ray fluorescence analysis with a pyroelectric X-ray generator, *Analytical Sciences* **20**, 1211–1215 (2004).

An X-ray tube provides the best performance of the sources currently available. This accounts for its use in all laboratory XRF instruments and increasing use in hand-held instruments. The use of this source is driven by the ability to do stratigraphic analysis in the borehole. Its main drawback is its complexity – an operating X-ray tube has never been flown in a spacecraft. The complexity is mainly due to the high voltage, which necessitates potting or other insulation in atmosphere and a high vacuum inside the X-ray tube itself. Generation of the high voltage consumes power, weight, and space. High voltage cabling may be required in the umbilical. All these considerations are challenging and potential failure points. However, the use of an X-ray tube is necessary for good analytical performance and it is only a matter of time before they come into routine use for planetary exploration. High vacuum tubes of similar construction are routinely used in deep space probes and communications satellites[16]. Much more will be known once the probe being constructed under this contract is operating and can be evaluated.

Final design (Dia. 26.9 mm)

The final design is shown in Figure 10. It is based on the above 3 designs and contains elements from all of them. The diameter is 26.9 mm (well below the 30 mm limit) and the length will be about 25 cm. The 3 mm reduction is a direct result of this study.
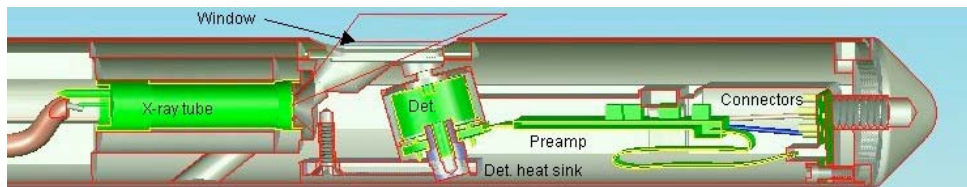

Figure 10. Final design (Dia. 26.9 mm)

Future possibilities – toward 10 mm

Because the detector is the size-limiting component, it should be the focus of any development effort aimed at reducing the size of the down-hole portion of the probe. The high voltage power supply would also benefit from some effort. Several vendors were optimistic about constructing a supply to fit within a 10 mm diameter. Typical potting compounds have dielectric strength of about 400 volts per mil (16 kV per mm). In a 10 mm diameter, only about 2 mm on each side would be required to stand off 30 kV, leaving 6 mm in the center for power supply components. Current miniature electronics

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[16] http://www.grc.nasa.gov/WWW/RT1998/5000/5620wilson1.html, verified Jan. 4, 2006.

and the construction of a Cockcroft–Walton voltage multiplier make such a power supply feasible.

None of the vendors were willing to speculate about making a smaller detector package, much less give any cost estimates. The authors of this study have considerable knowledge about and experience with this detector type in general and several specific units. The actual detector diode has dimensions of about 2.5 x 2.5 mm and is about 0.5 mm thick. It does not pose a significant size limitation. The cooler is larger and probably poses the main limitation. It must be situated between the diode and the mount, further restricting the size. However, there is no reason that a detector to fit within a 10-mm diameter probe could not be built. At least one vendor was willing to say that something could be done with enough funding. The authors estimate that a development contract with a suitable vendor would probably require $0.5–1M. This does not include the cost of X-ray source development to match the size reduction (about $100k) and the manpower to produce the specification, oversee the contract, and evaluate the resulting detector. Such an effort has a very high probability of success; there are no known impediments, other than cost, to achieving the required size.

Summary of Vendor Search

A broad search was conducted to identify any potential vendors of components that might aid in reducing the size of the new probe. To insure that no possibilities were overlooked, a letter was sent to a wide range of companies to request information. The list of companies was obtained from the *Physics Today Buyers Guide*, 2004–2005 Edition and from the exhibitor list at the Denver X-ray Conference held in Colorado Springs, CO, August 1–5, 2005. The list also included all of the vendors used to supply components for the SCAPS sensor and was augmented by any likely vendors known to the authors.

The letter was sent to 124 companies (the full list and the letter are available on request). Of these companies, 27 returned a written response to the inquiry. Many of these simply included product literature or letters giving the capabilities of the company. Six letters were returned by the postal service and several attempts to obtain recent addresses via web searches and directories failed. A few vendors of high voltage power supplies, at least two of which were good candidates for miniaturizing the necessary supplies for the probe, responded with "no bid." In the case where the reason could be determined it was because of limited manpower.

Eleven companies were identified as high-priority possibilities either because they had supplied components for the SCAPS sensor or manufactured specific X-ray sources or detectors that could be used to miniaturize the new probe. These companies were contacted by e-mail and telephone to obtain as much technical information as possible. It was at this stage that the reputations of the authors and their standing in this field were crucial. Several of these vendors were willing to reveal the details of their products and to discuss future plans for improvements. They were also willing to speculate about what could be done if development money were available and how much it would take to achieve specific targets of size and performance. The vendors were NOT willing to include this information in their written response. If fact, several of the priority vendors were not willing to make written responses at all other than what is available on their websites. The small size of the X-ray spectroscopy community made them wary of written comments. Because specific individuals were willing to talk to us, their names and the names of their companies are not identified here. References to products available on company websites are included where possible.

Conclusions

The size of the prototype instrument being built under this contract can be reduced from 30 mm to about 22 mm. This is possible with the new, miniature X-ray tubes being offered by several vendors. This smaller design uses available, off-the-shelf detector modules but requires the detector to be mounted axially. This will result in a minor reduction in performance and require the high voltage power supply to be placed up-hole. The likely performance cost will be a factor of two increase in MDL for sodium, with decreasing effects for the elements across that row in the periodic table (through chlorine). There would be little or no monetary cost for this change and little risk.

Improved techniques now allow the high voltage power supply to be included in the down-hole head for the 30-mm design. If the design is changed to reduce the size to 22 mm, the supply will not fit without some engineering effort (about $50k). The advantages of having the high voltage supply down-hole are the reduced size and complexity of the umbilical cable. This would make the umbilical easier to adapt to Mars ambient and autonomous deployment.

A dramatically reduced diameter, down to 10 mm, is possible but will require a serious development effort. There are no fundamental limitations to achieving 10 mm diameter, but the detector module will have to be completely re-designed. Further reduction below 22 mm is not possible without re-designing the detector. Some engineering of the X-ray source will also be necessary, but the cost is small compared to the detector effort. The authors estimate the cost of the required development effort at about $1M, and this is basically because there are no current market drivers to further reduce the size of the detectors, and thus the cost would have to be borne by this project. There is relatively little risk for this effort because it is mainly re-arranging and re-packaging of the same components.

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13. ABSTRACT *(Maximum 200 words)*

This report describes the recent work to design a borehole X-ray Fluorescence Spectroscopy (XRFS) instrument for the NASA Mars Subsurface Access Program. The instrument is to be used for elemental analysis of regolith strata in a pre-drilled borehole. The drills being developed now create a 30-mm diameter borehole; drilling a hole this size may require excessive power, so reducing the diameter of the borehole and probe would be a great advantage. This project began with a broad inquiry into the critical technologies for the components of the XRFS probe. The second part included a review and summary of the important considerations for the design of an XRFS borehole probe. Several preliminary designs were developed as the third component of the study. Finally, an evaluation of alternate sources for the primary X-rays was included. This evaluation covered the miniature X-ray tubes used in an existing probe and options for the proposed probe.

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